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EVALUATION OF SOLID LUBRICANTS: TEMPERATURE PROGRAMMED DESORPTION OF MoS₂ ON MOLYBDENUM AND OF ION-IMPLANTED MoS₂ ON MOLYBDENUM

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November 1988

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ABSTRACT

Thermal programmed desorption (TPD) is utilized in order to assess the lubricating properties of metal dichalcogenide films applied to a number of metal substrates. TPD spectra documenting desorption and decomposition products were analyzed for MoS₂ burnished on molybdenum metal with and without subsequent 86 kV nitrogen ion implantation. Desorption order, rate, energy, and the preexponential of evolved lubricant and nitrogen species from the molybdenum substrates were determined. The surface chemistry of heated lubricant-substrate system was investigated by means of X-ray photoelectron spectroscopy (XPS). The results presented clearly indicate that the mass spectroscopic TPD techniques developed for this investigation coupled with careful surface analysis yield critical in situ assessment of the capability and the operational limits of this and other solid lubrication systems.

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INTRODUCTION

The need for advanced materials for use in either high temperature and/or high vacuum and space environments have recently promoted significant research and development efforts ¹⁻⁷ in the area of solid lubrication technology. Although several metals, metal oxides, metal halides, metal sulfides, metal selenides, and other compounds and mixtures of compounds have been shown to exhibit good solid lubrication properties, metal dichalcogenides in general and molybdenum disulfide ⁸⁻¹⁰ in particular have emerged as the most promising candidates for solid lubrication in a number of important vacuum and space applications.

The structure of MoS₂,¹¹ shown in Figure 1, is characterized by a layer configuration which consists of a hexagonal layer of Mo atoms sandwiched between two planes of hexagonal sulfur atoms. The atoms are arranged so that the sulfur planes are staggered with respect to the plane of metal atoms allowing each Mo atom to be placed at the center of a trigonal prism formed by the surrounding six sulfur atoms. A consequence of this structure is that relatively weak forces between the sulfur-sulfur planes account for the excellent lubricating properties similar to those of graphite and other similar layered structures.

The deleterious effects of environmental conditions on the performance of these materials are two critical engineering parameters which have been examined by several tribologists ¹²⁻¹⁴ in order to optimize these compound's lubricity for specified application conditions. In this work we utilize the method of thermal programmed desorption (and/or decomposition) (TPD) to determine the desorption and/or decomposition kinetics of burnished and nitrogen ion- beam mixed MoS₂ films on molybdenum.

Thermal programmed desorption ¹⁵⁻¹⁷ is a technique in which the temperature of an adsorbed film or coating on a surface of interest is raised from an ambient or low temperature in a programmed manner, usually linearly with time, and the resulting desorbed, evaporated, or decomposed species are monitored and recorded by a suitable detector, usually a mass spectrometer. This process yields a spectrum of the rate of

evolution f various species from the surface as a function of surface temperature. Analysis of sets of TPD spectra yield kinetic information on the bonding of the film or coating to the surface, as well as provide insight into the processes occurring at the surface and leading to the evolution of the evaporated species.

The present work was undertaken in order to assess the usefulness of the TPD technique in the analysis of the high temperature degradation mechanisms of lubricating metal dichalcogenide films applied to various systems. A better understanding of such degradation mechanisms can lead to the development of improved preparation techniques, improved application techniques, and also the development of surface modification techniques capable of blocking particular degradation pathways.

In this report of phase one activity, results on the modification of a MoS₂ film by ion implantation are also presented. Ion implantation can introduce high energy, metastable states into the metal-film systems and in principle the presence of these metastable states can modify the properties of the lubricating film-metal system.

This phase of our effort focuses on the effect of temperature not only as a degradative process, but also as a diagnostic tool for investigating solid lubricant systems. In particular, TPD spectra are presented for: molybdenum metal; ion-implanted molybdenum metal; ion-implanted MoS_2 coated molybdenum metal; and MoS_2 coated molybdenum metal which was not ion implanted, but the MoS_2 was applied by various burnishing techniques.

EXPERIMENTAL

The TPD evaluation system shown in simplified form in Figure 2 has been developed in order to study the gaseous species evaporating at elevated temperatures from solid lubricant coated surfaces. It consists of a pumping chamber which includes a 400 l/s ion pump and a titanium sublimation pump and an experimental chamber separated from the pump by a gate valve. After baking overnight at 370 K, the base pressure was in the low 10^{-9} Torr range. The sample holder conveniently held three samples but could be used to hold several more. Each sample was positioned so that when heated the gaseous

species evaporating from the surface could enter the mass spectrometer ionizer by a line of sight path. The mass spectrometer is a Finnagan Model 1015 quadrupole mass spectrometer modified for molecular beam studies. Mass and transmission calibration was performed by introducing perfluorotributylamine which has a known fragmentation pattern.

Finely ground crystals of MoS₂ were applied to clean etched 0.002-inch-thick molybdenum metal foils. The lubricants were burnished onto a defined area of one side of the foil with a paper lab wipe, a nylon wiper, or a clean tantalum foil. About 100 micrograms cm⁻² MoS₂ adhered to the paper-rubbed metal surface as determined with a Cahn RH electrobalance. This amount of MoS₂ constitutes a coating containing 375 layers or 2300 Å thick. Chemical analysis indicates that the MoS₂ powder used in this investigation contains between 1.6 and 1.9% carbon (depending on the sample) and less than 0.3% oxygen for all samples. The amount of carbon impurity is equivalent to one carbon for every 3.8 to 4.6 formula units of MoS₂.

The coated foils were attached to specially designed holders. These holders enabled resistive heating of the metal foil to temperatures higher than 2000 K. A calibrated 3% Re/W-25% Re/W thermocouple pair was spot welded to the back of the sample for temperature measurement. The thermocouple signal was compared to a reference voltage and the difference was used to control the output of the heater power supply. The reference voltage was programmable and was adjusted to give a ramp rate of 7.5 degrees sec⁻¹.

A Teknivent Model 1050 mass spectrometer data system controlled the quadrupole, recorded ion currents and temperature, and initiated the heating of the sample. Experiments were first undertaken to survey the entire mass spectrum as the sample temperature was ramped. Next, the experiment was repeated and only those masses indicated by the survey were recorded, greatly increasing the signal to noise ratio. The lowest mass monitored was carbon at 12 amu. The high mass limit of the quadrupole was about 750 amu and this allowed the gaseous molybdenum oxide species $(MoO_3)_x$, (x = 1, 2, 3, 4) to be monitored.

X-ray photoelectron spectra (XPS) were taken for coated foil samples in both the survey and high resolution mode. The MTL Leybold-Hereaus LHS-12 Surface Analysis System was used to obtain these spectra. It includes a hemispherical electron energy analyzer, Mg and Al X-ray anodes, and a heated probe capable of heating samples to 950 K.

Some of the coated foils were ion-beam modified with nitrogen ions. The implantation was performed with the MTL Zymet system. An 86 keV ion beam consisting of nitrogen ions (N⁺ and 10% N₂⁺) was used. A 4 ma beam current was maintained for 92 minutes giving a total dose of 2 x 10^{+17} ions cm⁻². Foil samples were attached to an aluminum plate, and during the implantation the temperature which was measured with a thermocouple reached 177 °C \pm 17 °C.

A JEOL JSM-35C scanning electron microscope was used to study the physical appearance of the metal surface and the MoS₂ coated metal surface before and after ion implantation.

RESULTS

Electron Micrographs

A comparison of Figures 3 and 4 illustrates the smoothing and compacting effect of burnishing the MoS₂ crystals. From Figures 5 and 6, the surface modification caused by the ion implantation of molybdenum can be seen. A comparison of Figures 4 and 7 demonstrates the dramatic effect of the ion implantation process on the MoS₂ coating. Figures 8 and 9 show this dramatic effect at higher magnification, and clearly indicate surface reconstruction.

MoS₂ Coated Non-Implanted(C)

The decomposition of the coating starts as low as approximately 565 K (292 °C). This low temperature decomposition results in SO_2 vaporization. At higher temperatures both S_2 and CS_2 vaporize.

- 1. Low Temperature Mass 64 Peak (see Figure 10) Though both S_2 and SO_2 molecules have masses of 64, the isotope ratio between mass 64 and mass 66 indicates SO_2 instead of S_2 . The maximum rate of vaporization occurs near 700 K. The pc ak shape suggests second order kinetics with an activation energy for vaporization of 19 kcal mol⁻¹ and a pre-exponential factor of 1 x 10^{-10} .
- 2. High Temperature Mass 64 Peak (see Figure 10) Based on the isotope ratio and fragmentation pattern, this peak was found to represent vaporization of S_2 molecules. This mass 64 peak represents a major fraction of the total volatile sulfur species formed during coating decomposition. The peak begins at about 1140 K and reaches a maximum rate of vaporization at 1465 K. The peak shape indicates first order kinetics, with an activation energy of 81 kcal mol⁻¹ and a pre-exponential factor of 5 x 10^{11} .
- 3. High Temperature Mass 76 Peak (see Figure 10) The peak at mass 76 represents vaporization of CS_2 molecules. This peak is comparable in size to the mass 64 peak, but is more complex. Both the mass 64 and mass 76 peaks have their maximum rate of vaporization at 1465 K. In the area immediately surrounding this temperature, the mass 76 peak has nearly the same shape as the 64 peak. Features indicating smaller peaks begin to appear at about 150 degrees on either side of the main mass 76 peak. Analysis of the main peak indicates first order kinetics with an activation energy of 80 kcal mol⁻¹ and a pre-exponential factor of 2 x 10^{11} .
- 4. The Effect of Burnishing on the Mass 76 Peak (See Figure 11) Clean Ta metal which is not a source of carbon shows one main peak with a low temperature shoulder in the bottom panel of Figure 11. This low temperature peak is seen in the spectra for both the nylon and paper-rubbed samples. Paper rubbing induces a substantial high temperature CS₂ state as shown in the top panel of Figure 11. The nylon-rubbed sample shows peak broadening due to the presence of these two additional states as indicated in the middle panel of Figure 11.
- 5. Evidence for Interaction of H_2O with the MoS_2 Coating (see Figure 12) The top panel of Figure 12 shows the simultaneous production of H_2O and SO_2 . It is well known that water oxidizes MoS_2 and causes degradation of lubrication properties 14 .

6. Other Sulfur Species (See Figure 12) - Elemental sulfur veporizes to various polymers (S_8 , S_6 , ...). The MoS_2 coating vaporizes primarily as S_2 , however some S_4 is observed as illustrated in the bottom panel of Figure 12.

XPS Spectra

Recent enhancements to the MTL Leybold-Heraeus LHS-12 Surface Analysis System have enabled the acquisition of XPS spectra before and after heating the MoS₂/Mo sample to 450 °C. Figure 13 shows an XPS survey spectrum which was taken before any surface treatment, including heating, took place. Figure 14 shows the molybdenum 3d peaks before heating. The peak at 228.9 eV is interpreted as one of a pair of Mo 3d (IV) peaks. The peak at 232.1 is interpreted as the sum of Mo 3d(VI) and the Mo 3d(IV) lines. The peak at 235 eV is interpreted as one member of a pair of Mo 3d(VI) peaks. Based on these assumptions, a synthesized Mo 3d spectrum based on MoS₂, MoO₂, and MoO₃ is shown in Figure 14 along with the experimental points. The agreement is satisfactory.

Figure 15 shows a survey spectrum of the MoS₂ surface after heating to 450 °C. It should be noted that this temperature is high enough to desorb the water and SO₂. Figure 16 shows the Mo 3d peaks after heating. Note that the peak at 235 eV is now missing and the Mo 3d peak pair ratio more nearly corresponds to that for MoS₂. Figure 16 also shows a synthesized Mo 3d spectrum based on MoS₂ and MoO₂ along with the experimental points obtained after heating. The agreement is satisfactory.

MoS₂ Coated, Ion-Implanted Metal (CI)

The decomposition of the coated ion-implanted sample (CI) is distinctly different from that of the coated, non-implanted (C) sample. There is a marked reduction in the SO_2 peak, and the S_2 and CS_2 peaks are shifted to lower temperatures. The nitrogen implantation of molybdenum metal and MoS_2 coated molybdenum metal resulted in nitrogen vaporization as monitored at mass 14.

- 1. Low Temperature SO_2 Peak (see Figure 17) This peak with a maximum in the 700 K region is due to SO_2 vaporization. The relative magnitude of the ion-implanted SO_2 peak is greatly reduced compared to the non ion-implanted sample. Figure 17 shows a comparison between the SO_2 and H_2O peaks.
- 2. High Temperature S_2 and CS_2 Peaks (see Figure 18) The top panel in Figure 18 shows that the major mass 64 peak due to S_2 vaporization occurs at 1378 K. This is 87 degrees lower than the major peak of the coated non-implanted sample. On the low temperature side of the 1378 K peak, a smaller unresolved peak has been produced.

The mass 76 spectrum shown in the lower panel of Figure 18, due to CS_2 vaporization, is characterized by a combination of at least three peaks. The only resolved peak is the largest one which is found at 1378 K, precisely the same temperature as the major S_2 peak. As in the case of the 64 peak, on the low temperature side, there exists a smaller unresolved peak. On the high side of the CS_2 peak, slow tailing suggests another small unresolved peak.

3. Nitrogen Mass 14 Peak (see Figure 19) - The nitrogen vaporization from molybdenum metal and from MoS_2 coated molybdenum metal is shown in the top panel of Figure 19. In both cases the spectra show that the nitrogen is released from discrete states. For the metal alone, a large peak at 1248 K and a small peak at 1413 K are observed. For the coated sample the main peak occurs at 1428 K. It may be noted that the presence of the coating has caused a 180 degree increase in the nitrogen release temperature.

The lower panel of Figure 19 shows that a large fraction of the sulfur is released before the nitrogen release reaches its maximum. One may also note the simultaneous release of nitrogen and sulfur for the minor states occurring at approximately 1050 K.

DISCUSSION

- 1. The Low Temperature SO₂ Peak (see Figures 10, 12, and 17) The low temperature SO₂ peak shown in Figures 10, 12, and 17 arises from a desorption process which obeys second order kinetics and appears to be related to the presence of water. Since the Mo 3d XPS spectrum indicates that some of the Mo(IV) was oxidized to Mo(VI) on the surface, one possible mechanism for the production of SO₂ may involve the reaction between the MoS₂ and oxygen containing species. The area of the low temperature SO₂ TPD peak is 20 % of the area of the high temperature S₂ peak as shown in Figure 10. This comparison indicates that SO₂ production can be an important pathway leading to lubricant failure. In contrast, Figure 17 shows that ion implantation has reduced the low temperature SO₂ peak by more than a factor of 10. Therefore, the importance of this pathway to lubricant failure may also be reduced by ion implantation of the lubricant.
- 2. The High Temperature Sulfur Peak Figure 10 summarizes the TPD results for the MoS_2 film on molybdenum metal (with no ion implantation). The dominant feature shown in Figure 10 is the first order S_2 peak. This S_2 peak arises from the thermal decomposition of the MoS_2 film. The decomposition process starts at 1140 K and reaches a maximum rate at 1465 K. Clearly, the onset of decomposition sets an upper temperature limit for long term lubrication applications.

Figure 7 shows that the onset of CS_2 vaporization starts at 1128 K and reaches a maximum at 1465 K. The main part of this CS_2 peak is similar in shape to the first order S_2 peak. This similarity suggests that both S_2 and CS_2 share a common rate determining process in their formation. The CS_2 peak is roughly equal in area to that of the S_2 peak. This suggests that a large carbon impurity is present in the film-metal system. The XPS spectra and elemental analysis confirm the presence of carbon as an impurity in the MoS_2 .

Figure 18 summarizes the TPD results for the ion implanted molybdenum metal- MoS_2 film system. The main S_2 and CS_2 peaks are shifted to lower temperatures by

87 degrees. Figure 18 also shows that ion implantation dramatically reduces the thermal stability of the MoS₂ coating, because the film vaporization rate is increased for temperatures between 1150 and 1350 K.

The lower panel of Figure 19 shows that ion implantation has induced a minor peak at 1048 K. Figure 19 also indicates that there is a corresponding minor nitrogen peak at the same temperature.

3. The Nitrogen Peaks - The TPD spectrum for ion-implanted bare metal is shown in Figure 19. This spectrum is characterized by one major peak at 1248 K and one minor peak at 1413 K. These peaks may arise from nitrogen trapped in metal defect sites such as interstitials or vacancies. The interpretation of these peaks as originating from a nitride phase(s) is being considered.

Figure 19 shows that the main nitrogen peak for the ion-implanted MoS₂ coated system is shifted to a higher temperature by 180 degrees. There remains a small peak at 1258 K which may result from the same state of nitrogen observed at 1248 K for nitrogen implanted bare metal.

It is worth noting that the presence of the MoS₂ coating changes the main nitrogen peak to a higher temperature state. This shift to a higher energy state could result in improved system performance. At this time, the mechanism for the preferential filling of the high energy state is unclear, but this should be investigated in the future.

CONCLUSIONS

The results presented in this phase one report clearly indicate that the mass spectrometric TPD techniques developed for this investigation can produce a wealth of new information about the thermal and chemical stability of thin solid lubricant films. In particular, TPD is sensitive to the film burnishing technique, as well as to the effect of ion-implantation. These results strongly suggest the TPD may contribute to our understanding of the thermal and chemical stability of thin films applied by other techniques such as sputtering and ion plating.

In the future, we hope to continue our joint program and to use our combined TPD and XPS techniques to better understand various properties of coating systems of importance to selected Army missions.

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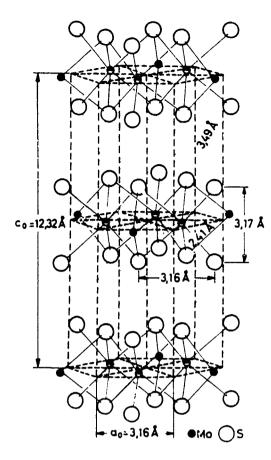


Figure 1. Crystal structure of molybdenum disulfide.

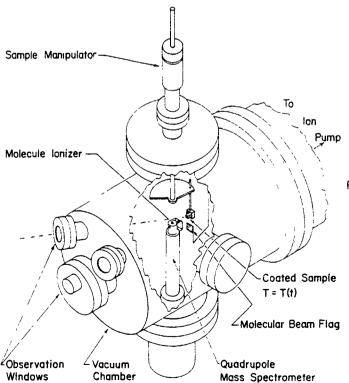


Figure 2. System for the evaluation of the thermal and chemical stability of solid lubricants.

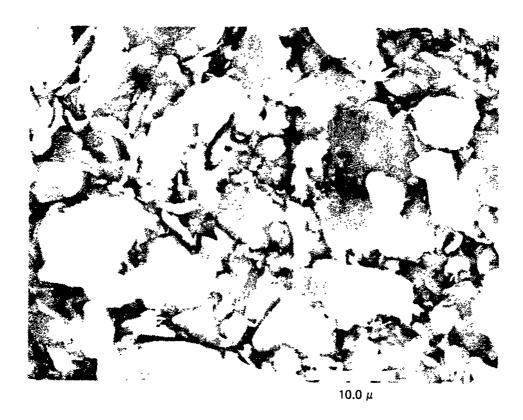


Figure 3. MoS_2 powder (25 kV, Mag. 2000X).



Figure 4. MoS_2 coated molybdenum metal (MoS_2/Mo) (CM) (Mag. 2000X).

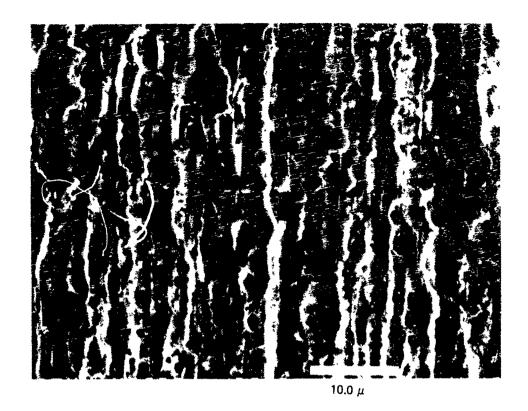


Figure 5. Molybdenum metal (M) (25 kV, Mag. 2000X).

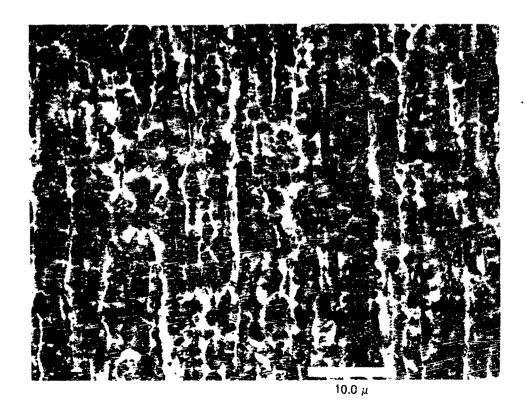


Figure 6. Ion-implanted molybdenum metal (IM) (25 kV, Mag. 2000X).

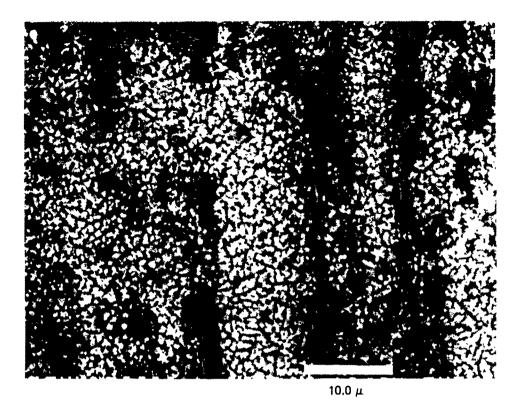


Figure 7. Ion-implanted MoS_2/Mo (ICM) (25 kV, Mag. 2000X).



Figure 8. MoS_2/Mo (CM) (Mag. 6000X).



Figure 9. Ion-implanted MoS_2/Mo (ICM) (25 kV, Mag. 10,000X).

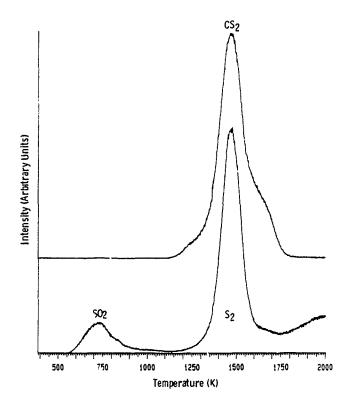


Figure 10. Intensity (desorption rate) of ${\rm SO}_2$, ${\rm S}_2$, and ${\rm CS}_2$ as a function of temperature for ${\rm MoS}_2({\rm burnished})/{\rm Mo}$ (CM).

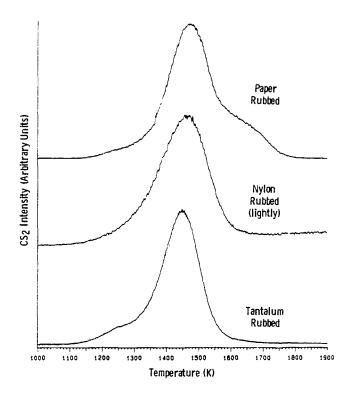


Figure 11. Intensity (desorption rate) of ${\rm CS_2}$ as a function of temperature for ${\rm MoS_2(burnished)/Mo~(CM)}$ for different burnishing techniques.

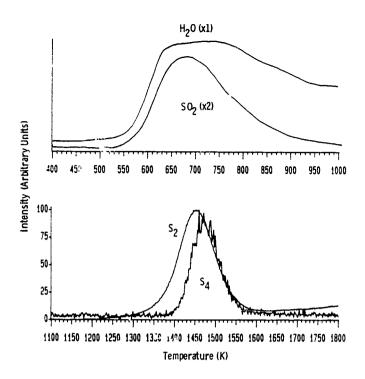


Figure 12. Top \sim intensity (desorption rate) of H₂O and SO₂ as a function of temperature for MoS₂(burnished)/Mo (CM); bottom - intensity (desorption rate) of S₂ and S₄ as a function of temperature for MoS₂(burnished)/Mo (CM).

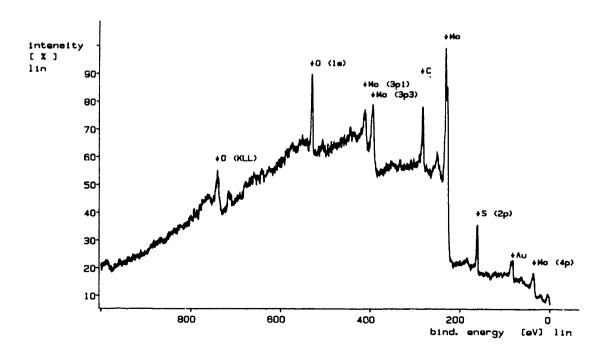


Figure 13. XPS survey spectrum for MoS_2/Mo (CM) before heating.

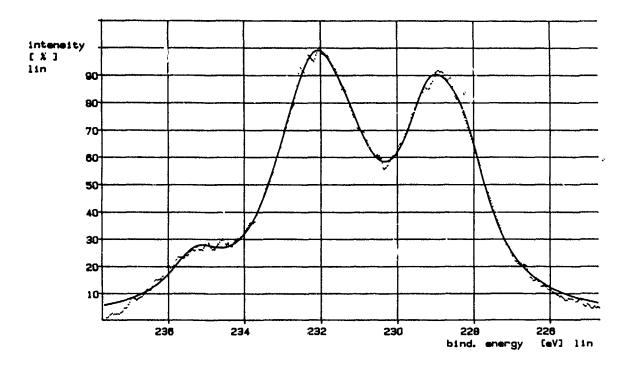


Figure 14. Synthesized Mo 3d spectrum (solid line) and the experimental Mo 3d spectrum (points) before heating.

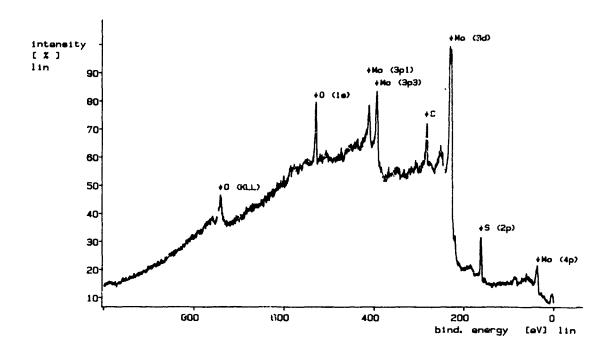


Figure 15. XPS survey spectrum for MoS_2/Mo (CM) after heating to $450^{\circ}C$.

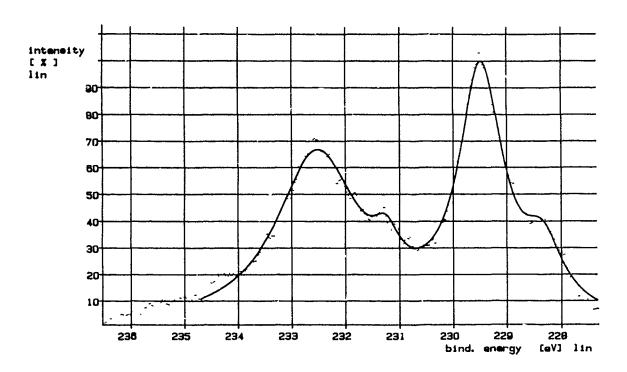


Figure 16. Synthesized Mo 3d Spectrum (solid line) and the experimental Mo 3d spectrum (points) after heating to 450° C.

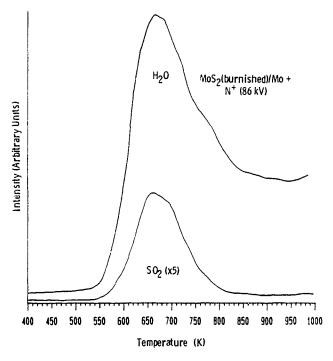


Figure 17. Intensity (desorption rate) of $\rm H_2O$ and $\rm SO_2$ as a function of temperature for ion-implanted $\rm MoS_2/Mo$ (ICM).

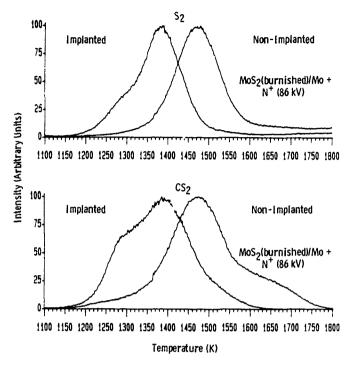


Figure 18. Top - changes in the S $_2$ release due to ion implantation for the MoS $_2$ /Mo system; bottom - changes in the CS $_2$ release due to ion implantation for the MoS $_2$ /Mo system.

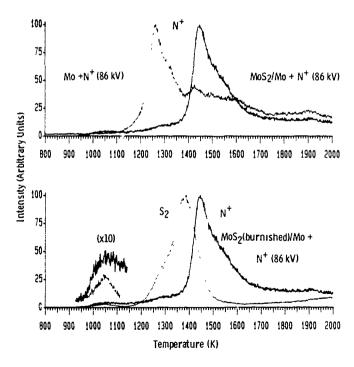


Figure 19. Top - comparison of the nitrogen release from N $^+$ implanted metal (IM) and from MoS $_2$ /Mo (ICM); bottom - comparison of the S $_2$ release with the nitrogen release for N $^+$ implanted MoS $_2$ /Mo (ICM).

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